

## Historic, archived document

Do not assume content reflects current scientific knowledge, policies, or practices.



732  
2 Ag 82

AIC-373

June, 1954

# X IN-PACKAGE DESICCATION OF DEHYDRATED FOODS X

✓  
Carl E. Hendel and Horace K. Burr ✓



800 Buchanan St.,  
Albany 6, California

Western Utilization Research Branch of Agricultural Research Service  
UNITED STATES DEPARTMENT OF AGRICULTURE

## Table of Contents

Page no.

Survey of Literature - - - - -	2
Effects of In-Package Desiccation - - - - -	2
Selection of Desiccant - - - - -	3
Selection of Desiccant Container - - - - -	4
Date of Desiccation - - - - -	5
Summary - - - - -	7
Appendix: Tests for Calcium Oxide and Containers - -	7
I. Tests of Expansion of Calcium Oxide Desiccant during Hydration and on Volume After Hydration -	8
II. Tests of Rupture-Proofness of Desiccant Container - - - - -	9
III. Test of Calcium Oxide Desiccant Capacity and activity - - - - -	11
Literature Cited - - - - -	12

Cover: The photograph on the cover shows a no. 10 can of dehydrated cabbage and stretchable net of calcium oxide, used as desiccant.

## In-Package Desiccation of Dehydrated Foods

Carl E. Hendel and Horace K. Burr

In-package desiccation is a process that reduces moisture content of a product after it has been packaged. The product is packaged together with a small package of drying agent, and drying occurs during storage. Moisture is slowly transferred from the product to the desiccant, which is held in a moisture-permeable container.

By the process of in-package desiccation, dehydrated foods can be dried to substantially lower moisture levels, without heat damage, than is commercially feasible by other methods. Quality retention in many dehydrated foods is markedly improved by this attainment of lower moisture levels.

Current interest in desiccation within packages has been aroused in part by recently renewed interest in vegetable dehydration, and in part by the development at the Western Utilization Research Branch of vacuum "puff-dried" citrus-juice powders. These powders are promising new products, which require in-package desiccation if they are to be economically dried to moisture levels at which they will have satisfactory storage stability (35).

In-package desiccation of dehydrated foods was first studied at this Laboratory during World War II. This work was an outgrowth of comprehensive storage studies in which quantitative measurements showed the marked effect of moisture content on the nonenzymatic browning of dehydrated vegetables (17,18,19). The program had been undertaken in response to Quartermaster needs for control of browning during warm and tropical storage. It was found that by in-package desiccation, dehydrated vegetables could be made very resistant to browning.

This development was announced by L. B. Howard early in 1945 (11,12,13). Developmental work and small-scale commercial trials were quickly conducted by this Laboratory and by the Quartermaster Subsistence Research and Development Laboratory, the forerunner of the present Quartermaster Food and Container Institute for the Armed Forces. This cooperative work was highly successful, and resulted in a military specification (37) for what was called "in-can dehydration". This specification came just at the end of the war and was not used for regular military procurements.

Since World War II certain further work on in-package desiccation of dehydrated foods has been conducted at this Laboratory. The present report briefly summarizes both earlier and more recent studies of (a) protection obtainable for various products by in-package desiccation, (b) choice of desiccant, (c) choice of desiccant container, and (d) rate of desiccation. An appendix gives empirical tests which have been used for the appraisal of suitability of calcium oxide samples and for the testing of potential desiccant containers.



## Survey of Literature

The principle of desiccant packaging has been known for many years. A considerable number of patents and publications exist. The objective, however, has usually been to prevent corrosion or to prevent increase in moisture content of a product from transfer of moisture through the container, rather than actually to dry the product.

The first record of desiccant packaging appears to be that of an English patent granted to W. T. Yule in 1845 (41) for "keeping animal and vegetable matters dry, when preserving them in closed vessels, by placing chloride of calcium or other chemical absorbent in such vessels, but not in contact with the matters to be preserved." Patents were granted to Ohlhaber in 1911 and 1912 (32, 33, 34) for the packaging of dry yeast with a desiccant. Other patents (1, 21, 23, 24, 26, 27, 28, 31) issued before World War II were for containers, and for drying agents, for the desiccant packaging of metal goods, of pharmaceuticals, and of foodstuffs such as cereal products, crisp bakery goods, candy, pepper, and coffee. During World War II, desiccant packaging was used on a large scale (9, 10, 40) by the Armed Forces in the Method II packaging of instruments, equipment, and machinery for maintenance of the humidity below that at which metals rust. In the past five years a number of publications (8, 15, 20, 25) have appeared on the desiccant packaging of a range of food products, and in 1950 a patent (3) was issued to Cotton and Schroeder (assigned to National Research Corp.) for application of the process to citrus juice powders.

The desiccants most commonly specified in these publications and patents are the physical-adsorption type (e.g., silica gel, alumina, and montmorillonite). Such desiccants have high moisture adsorptive capacity at the moderate relative humidities that are effective in preventing moisture absorption of many foods. Desiccants of this type, and also calcium sulfate, are listed in the citrus-powder patent of Cotton and Schroeder; however, such desiccants have low capacity at the low humidities necessary for the efficient continued drying of foods that have been dehydrated. Calcium chloride is specified as the desiccant in a number of the early patents, but even this drying agent has but moderate capacity in the relative humidity range of 1 to 5 percent, needed for adequate desiccation of some dehydrated foods. Minimizing the sacrifice of container volume to the desiccant is of course important. For this reason, calcium oxide was selected for most of the in-package desiccation studies at this Laboratory. A relatively small amount of this drying agent can effectively desiccate dehydrated foods, since it has full capacity at very low relative humidities. The problem of expansion of the calcium oxide on hydration is important, and a considerable amount of work has been necessary to develop desiccant containers which will withstand this expansion.

### Effects of In-Package Desiccation

Extensive data on the effects of in-package desiccation on dehydrated vegetables have been reported by R. R. Legault and coworkers (16). In-package desiccation provided substantial protection against loss of ascorbic acid and loss of sulfite, as well as nonenzymatic browning. At least 180 days at 100° F. were required for even a "low" degree of browning in the dehydrated

cabbage, onion, white potato, and sweetpotato for both air and nitrogen packs. This finding is considered significant, since the "low" degree of browning approximates the amount barely detectable to the eye; thus the product differed but little from the original at this stage. Acceptability for 180 days at 107°F. is a stability goal of the Quartermaster Corps for dehydrated vegetables and other products.

The dehydrated carrot received less protection than other products, as a result of very slow desiccation (see later section on "Rate of Desiccation"). In one lot of sweetpotato, packed in air, fading of color and development of oxidative off-flavor were accelerated by in-package desiccation. These effects were not observed with the five other vegetables (white potato, carrot, cabbage, beet, and onion) (16), and they have not occurred in orange-juice powder. They of course did not occur in sweetpotato packed in nitrogen. For certain products, packing in an oxygen-free atmosphere may prove desirable in conjunction with in-package desiccation.

The rate of oxygen absorption by air-packed dehydrated vegetables was decreased at 120°F., but was increased at 75°F. At 100°F. in-package desiccation retarded absorption of oxygen by beet, onion, sweetpotato, and cabbage, while it accelerated oxygen uptake by sulfited white potato. The effect was very slight, if any, in carrot and non-sulfited white potato.

In-package desiccation has proved effective in preventing caking of dehydrated fruit and vegetable powders. With dehydrated onion powder this prevention of caking was demonstrated in a cooperative experiment by the J. R. Simplot Co. of Caldwell, Idaho, and the Filtrol Corporation of Los Angeles, Calif.; this was briefly described in an article by Roger A. Lovett (20) and more extensively in an unpublished report by Ray L. Dunlap of the Simplot Company. Protection against caking and chemical deterioration of citrus-juice powder is reported in the patent by Cotton and Schroeder (3).

For the citrus-juice powders recently developed at this Laboratory, control of nonenzymatic browning and of off-flavor development, as well as prevention of caking, have been reported briefly by Strashun and Talburt (35), and in greater detail by Mylne and Seamans (29).

#### Selection of Desiccant

Because of its exceptionally high moisture absorptive capacity at low relative humidity, active calcium oxide is considered the preferred desiccant for dehydrated fruit and vegetable products. Figure 1 shows absorptive capacities of various commercial physical desiccants and of pure calcium chloride and calcium oxide. Variations between samples of these desiccants occur, but the data illustrate the relative humidity ranges at which various desiccants will be most useful.

---

1/ These effects were not observed in a lot of sweetpotato recently stored in air in this Laboratory by F. E. Lindquist and coworkers. Perhaps variety or cultural conditions affect the relation between moisture content and oxidative effects in sweetpotato and possibly in other commodities. Further work on the point is needed.



Since dehydrated fruit and vegetable products have very low vapor pressure at the end of desiccation--corresponding to relative humidities of 1 to 3 percent in some cases--a given volume of calcium oxide will absorb a considerably larger quantity of water from the dehydrated product than will the other desiccants. Hence a smaller sacrifice of container volume is necessary with calcium oxide as the desiccant.<sup>2/</sup>

For certain dehydrated vegetables such as cabbage and onion, particularly for civilian use, desiccants other than calcium oxide may have application, provided some further sacrifice in container volume to the desiccant can be permitted. Burr and Silveira (2) have successfully used montmorillonite (a hydrous aluminum silicate) on a laboratory scale for the in-package desiccation of dehydrated cabbage.

Calcium chloride has a high capacity at relative humidities from 5 to 14 percent, but only moderate capacity at lower humidities. Also it is deliquescent, forming a liquid at humidities above about 30 percent. For these reasons it has not been used in our studies.

Calcium oxide samples differ widely in their capacity and in their reactivity. Recalcined hydrated-lime samples have usually had higher capacity and higher reactivity than samples of calcined limestone. Lower volumetric expansion on hydration has also been observed with recalcined hydrated limes. An empirical test of calcium oxide desiccant capacity and reactivity is given in an Appendix (page 11).

Certain precautions in use of calcium oxide as the desiccant are necessary. This desiccant expands with great force during hydration; the desiccant container must permit the expansion or the lime will break the desiccant container. The volume of increase observed for various samples has ranged from about 60 percent to 170 percent of original volume.

Also, heat is evolved by lime as it hydrates. The amount of heat liberated is greater than that absorbed by the product as the water vapor is desorbed from it--16,700 calories per mole of water absorbed by the lime as compared to approximately 10,800 calories per mole of water vaporized from the product. It is therefore possible that large stacks may heat enough to cause damage to the product. It is not yet known if this will be a problem. If it is, provision for ventilation of large stacks may be necessary to prevent overheating.

#### Selection of Desiccant Container

The desiccant container must be sift-proof, must be strong enough to resist abrasion during handling, and must permit expansion of the calcium oxide if this desiccant is used. Three types of desiccant containers are satisfactory if properly selected and properly used.

---

<sup>2/</sup> No information is available as to the attitude of State or Federal regulatory agencies on the use of calcium oxide for desiccation of food products.



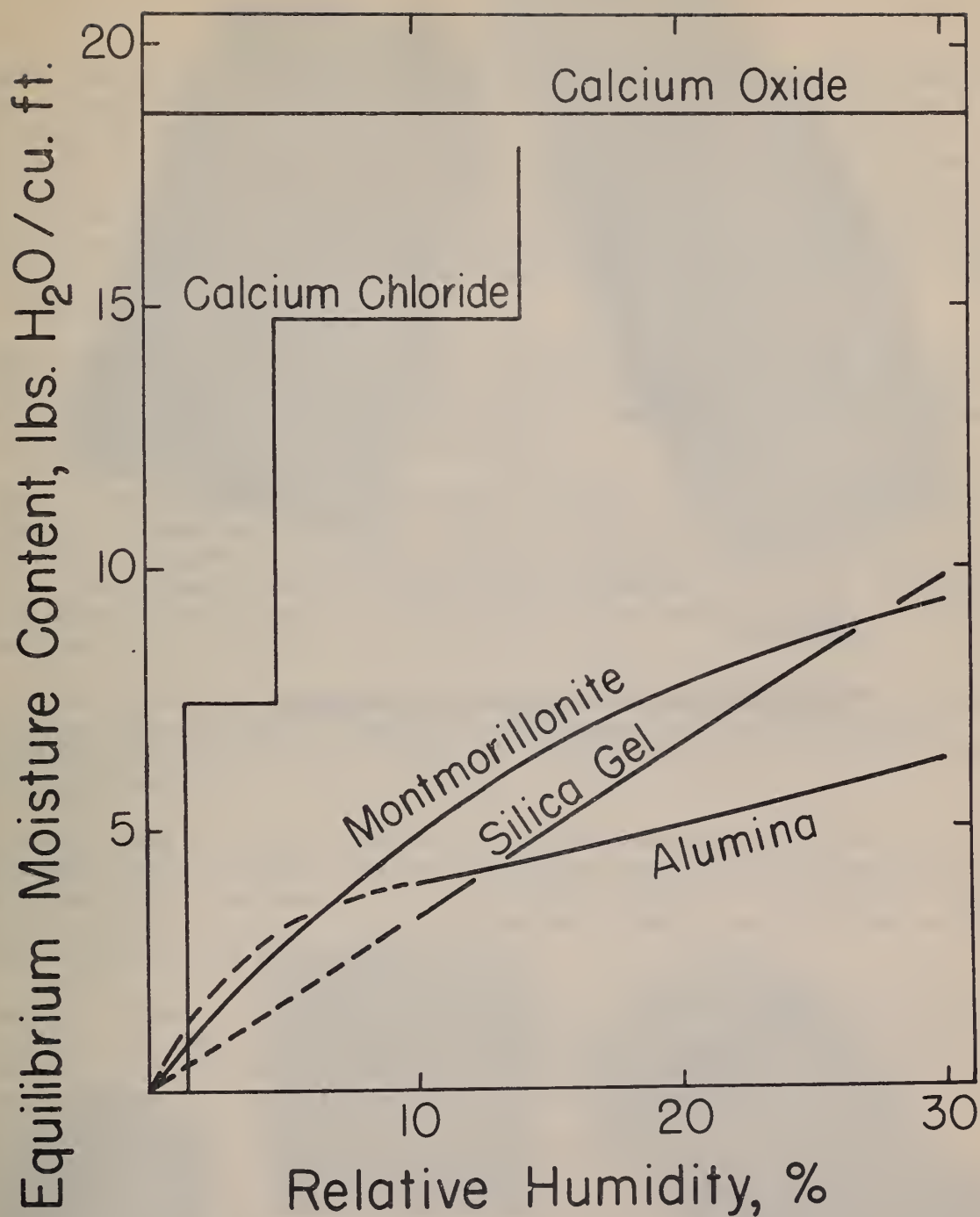


Figure 1.

Moisture absorptive capacities of calcium oxide (31), calcium chloride (31), montmorillonite (21), silica gel (4), and alumina (5) as functions of relative humidity. Based on bulk densities (lbs./cu. ft.) of 58 for calcium oxide, 45 for calcium chloride, 60 for montmorillonite, 50 for silica gel, and 45 for alumina.



Figure 2.

Non-stretchable desiccant containers.



Figure 3.

Stretchable heat-sealing desiccant bags.

One is the bag-type container with sufficient strength to resist rupture. Paper-coated jean cloth bags meeting the requirements for Military Specification MIL-D-3464 (39) appear to be the most promising of the non-stretchable bags tested. Heavy (18-oz.) canvas is satisfactory, but expensive. Jean cloth bags with separate Kraft paper liners were selected by the Quartermaster Subsistence Laboratory for the "in-can dehydration" specification (37). Ample volume to permit expansion of the calcium oxide is essential; otherwise the container may burst. Figure 2 shows two kinds of bag desiccant containers.

Rigid containers only partly filled with desiccant can be used. The calcium oxide expands into the unfilled part of the container. Figure 2 shows a rigid chip-board (cardboard) cylinder with metal ends, intended for use in 5-gallon cans of dehydrated vegetable.

A third type of container is produced from heat-sealing, stretchable bag materials. Examples of various sizes are shown in Figure 3. These bags are made of "Promset 831X",<sup>3/</sup> a product of the Mid-States Gummed Paper Co. This material has a two-way stretch which increases its ability to permit expansion of the calcium oxide desiccant without rupture of the desiccant container.

In tests to date the dehydrated product has settled enough during storage to permit the expansion of calcium oxide packed in bags; hence smaller sacrifice of container volume is necessary with bags than with rigid desiccant containers.

With desiccant bag containers, of either nonstretchable materials or of the stretchable materials available thus far, care should be taken that any unfilled part of the bag is not "pinched-off" from the filled part. Calcium oxide tends to "bridge" on hydration and as a result does not flow readily into unfilled parts of the bag.

Powdered calcium oxide samples have expanded somewhat less, and have exhibited lower tendency to rupture pinched-off bags, than have comparable samples in pellet form. A test of rupture-proofness of desiccant bags, with or without pinching off, is given in the Appendix.

#### Rate of Desiccation

In commercial practice it will often prove desirable to locate the desiccant within the package in such a way as to promote rapid drying of the product. During desiccation, water vapor must be transferred in succession through each of the package components, which are:

The individual product piece. The moisture must be transferred from the interior of each piece to its surface.

---

<sup>3/</sup> Mention of commercial names of materials does not constitute a recommendation by the Department of Agriculture over other comparable products not named.



The package atmosphere. The water vapor must diffuse from the surface of the piece, through the package atmosphere to the desiccant container.<sup>4/</sup>

The desiccant container. The water vapor must pass through the desiccant container, and

The desiccant itself. The moisture must be absorbed by the desiccant.

Each of these components offers some resistance to the moisture transfer, tending to slow it down. The individual resistances of these various components have been measured, for certain representative conditions, and the results have been expressed in terms of "transfer coefficients".<sup>5/</sup> From these transfer coefficients the effects of the individual package components have been estimated. The details will be reported elsewhere. The estimates show that little resistance is offered by active calcium oxide or by suitable desiccant containers.

Resistance of the package atmosphere can be large. The package atmosphere offers a very large resistance in containers of 10- to 15-inch depth (for example, in 5-gallon cans) if the desiccant is at one end of the container. This fact was demonstrated with containers of various depths. For dehydrated cabbage, 6.5 days were required to reduce the moisture content from 4 percent to 2 percent when the product depth was 3 inches (desiccant at one end), 13 days when the depth was 6 inches, and 25 days when the depth was 12 inches. For onion flakes the numbers of days required to reduce the moisture from 3.9 to 1.9 percent were, respectively, 10, 23, and 59 for product depths of 3, 6, and 12 inches. The beneficial effect of reducing the transfer distance through the package atmosphere is thus evident.

Some products can themselves offer substantial resistance. For carrot of  $3/8 \times 3/8 \times 3/16$ -inch size before drying, 41 and 65 days, respectively, were required for product depths of 3 and 6 inches in reducing the moisture content from 7.1 to 4.1 percent, the relatively small difference in time requirement being due to the resistance of the carrot itself. For this product the rate of desiccation was increased about 2- to 3-fold by a decrease in piece thickness from  $3/16$  inch to  $1/16$  inch. For orange-juice powder at product depths of 3 and 6 inches, 41 and 76 days, respectively, were required to reduce the moisture content from 2.7 to 0.7 percent. The studies mentioned above were conducted at 1 atmosphere of pressure. The storage temperature was 93°F.

---

<sup>4/</sup> It is recognized that in traveling from the product piece to the desiccant container a given water molecule may rarely remain continuously suspended in the gas phase. Rather it may be successively adsorbed and desorbed by a series of product pieces in moving from the piece where it originated to the desiccant container.

<sup>5/</sup> A part of this work was performed by the senior author during graduate study at the University of California, Department of Chemistry and Chemical Engineering, under the direction of C. R. Wilke.

The effect of temperature has been determined in another study (7). It was found that each 18°F. (10°C.) of temperature increase approximately doubles or triples the rate of desiccation of dehydrated vegetables

### Summary

Four aspects of in-package desiccation have been reviewed:

The protection obtainable by in-package desiccation. The process can provide protection against nonenzymatic browning of dehydrated foods, against the development of off-flavors in orange juice powder, against the caking of fruit and vegetable powders, and against losses of sulfite and of ascorbic acid.

Oxygen absorption by air-packed dehydrated vegetables was decreased at 120°F. but was increased at 75°F. For one lot of air-packed sweetpotato stored at 75°F., in-package desiccation accelerated fading of natural pigment and development of oxidative off-flavor. This effect was not observed with air-packed cabbage, onion, white potato, carrot, or beet, or with orange juice powder. Further study of this effect is needed. The results suggest that for some products best protection can be obtained by in-package desiccation in combination with packing in an oxygen-free atmosphere.

The desiccant. Because of its high water vapor capacity at low relative humidity, active calcium oxide is a preferred desiccant for dehydrated fruit and vegetable products.

The desiccant container. Several types of containers are suitable. If the desiccant is calcium oxide, the ability of the container to permit the expansion should be carefully tested.

The rate of desiccation. The package atmosphere can offer a very large resistance to moisture transfer, and hence can greatly retard the desiccation.

Some dehydrated fruit and vegetable products have a large resistance to moisture transfer; others do not, depending in part on their moisture content.

Calcium oxide desiccants and desiccant containers--if properly selected--will offer but small resistance to the moisture transfer.

### APPENDIX: Tests for Calcium Oxides and Containers

During the course of the in-package desiccation research at this Laboratory, a number of procedures have been devised to test the moisture-absorbing capacity of calcium oxide samples, the expansion of such samples during hydration, and the rupture-proofness of desiccant containers. These tests are empirical. Most of them are rapid, designed as tools to aid in the development or in the control testing of desiccants and desiccant containers. It is recognized that they are based on an arbitrary choice of conditions and that modifications may offer advantages; however, they may serve as useful guides to others who undertake either the use or the further development of in-package desiccation.



# I. Tests of Expansion of Calcium Oxide Desiccant During Hydration and of Volume After Hydration

Two tests have been used. Of these, the second is preferred.

Test 1. The volume of a known weight of calcium oxide is measured in a graduated cylinder. The calcium oxide is transferred to a second (shallow) container. It is then hydrated over saturated sodium bromide solution. After hydration the desiccant is again transferred to the graduate for measurement of its volume. The graduate has usually been tapped to constant volume. Error due to change in volume of the hydrated desiccant during transfer to the graduate can occur.

The weight of calcium oxide has usually been 100 grams. The desiccator can be at atmospheric pressure or it can be evacuated to increase the speed of hydration. At atmospheric pressure, 6 to 8 weeks may be required for hydration. Hydration by exposure to the open atmosphere is not satisfactory; absorption of carbon dioxide and formation of calcium carbonate may lead to serious error.

Test 2. The preferred test is rapid, requiring about 2 to 3 days, and the transfer of the desiccant from the graduate to the second container is avoided.

- a. Weigh 100 grams of the calcium oxide into a 250-ml. graduated beaker.
- b. Read the volume.
- c. Place the beaker in a No. 10 can cut to a height of about 3 inches. Add saturated sodium bromide solution to the can around the beaker to a depth of about 2 cm. Place the can in a desiccator that has no desiccator plate and contains about 2 cm. of saturated sodium bromide solution. The bottom of the can is immersed in the solution in the desiccator to permit rapid dissipation of the heat of hydration.
- d. Evacuate the desiccator.
- e. After hydration is complete, read the volume of the hydrated calcium oxide.

The course of desiccation can be followed, if desired, by occasional weighing of the beaker and its contents. The volume of desiccant has usually been read after compacting to constant volume by gentle tapping of the beaker on the table top.

The pressure in the evacuated desiccators has been 6 to 8 cm. of mercury; evacuation has been accomplished with a water aspirator.

Saturated sodium chloride solutions instead of saturated sodium bromide solutions have given satisfactory results. The relative humidity over a saturated solution of sodium chloride is about 75 percent; that over a saturated sodium bromide solution is about 55 percent. Hydration over pure



water is not satisfactory; the amounts of water absorbed can be greatly in excess of that corresponding to the hydration of calcium oxide to calcium hydroxide.<sup>6/</sup>

The test does not measure very accurately the volume the hydrated desiccant would occupy in a container of dehydrated food because no weight is pressing on the calcium oxide during hydration. However, it appears to be the best test presently known.

Attempts to hydrate calcium oxide with a weight resting on it during hydration have been unsuccessful. In several tests, weights of about 50 grams per square inch<sup>7/</sup> (with appropriate screen supports) have been placed on 100-gram calcium oxide samples in 250-ml. graduated beakers, but in most of the samples so tested, the bottom has been broken from the beaker by the force of the expanding desiccant. Breaking of the bottom from the beaker resulted from formation in the early part of the hydration of a hydrate "bridge" across the upper part of the beaker; this "bridge" proved stronger than the beaker when the calcium oxide in the lower part of the beaker expanded on hydration.

The breaking of thick-walled graduated beakers by the expanding calcium oxide is one of several demonstrations of its tendency to bridge during hydration. As a result of this bridging the calcium oxide does not flow readily during hydration.

## II. Tests of Rupture-Proofness of Desiccant Container

The following tests of the ability of desiccant bags to withstand the expansion of the calcium oxide can be applied with or without "pinching-off" the unfilled part of the bag.

Test 1. Rapid empirical test. To test under pinched-off conditions, the bag is picked up by one end to permit the calcium oxide desiccant to fall to the other end. The bag, with the unfilled part in approximately the same plane as the filled part,<sup>8/</sup> is placed on the bottom of a No. 10 can cut to about 3-inch height, and 3 pounds of sand are poured into the can, on the desiccant bag. The sand pinches off the unfilled part of the bag. The can is set in a desiccator containing saturated sodium chloride solution, and the desiccator is evacuated (residual pressure, 6 to 8 cm. of mercury). The

---

<sup>6/</sup> The abnormally high weight increase of calcium oxide over pure water is evidently due to formation of a hydrate of the hydroxide. Over saturated sodium bromide solutions the weight increase of commercial limes will be a little below the theoretical value of 32.1 percent, but over pure water the weight increase may be as much as 50 percent higher.

<sup>7/</sup> Fifty grams per square inch is the approximate weight per unit area on the bottom of a No. 10 can containing 3 pounds of orange juice powder (the quantity packed in this size of can).

<sup>8/</sup> As a somewhat more severe test, the unfilled part of the bag has sometimes been folded under the filled part.

desiccator plate has been removed and the bottom of the can is immersed in the saturated sodium chloride solution.

To test without pinching off, the bag is placed on the bottom of the No. 10 can with the desiccant spread somewhat uniformly across the bottom of the bag. The unfilled part of the bag is then readily available to the expanding desiccant.

The 3 pounds of sand exerts a force on the desiccant bag equivalent to that exerted on the desiccant bag in a No. 10 can of orange juice powder when the can is filled, sealed, and subsequently handled in such fashion that the desiccant container is at the bottom of the can during storage.

Removal of the desiccator plate and immersion of the No. 10 can in the sodium chloride solution is necessary if overheating of the calcium oxide and desiccant bag is to be avoided. In the first tests, the cans were placed on the desiccator plates, out of contact with the solution. The calcium oxide became so hot during the subsequent hydration that the seals of heat-sealed bags separated; moisture transfer was rapid and heat transfer very slow in the evacuated desiccators. Overheating is avoided by immersing the lower part of the cans in the sodium chloride solutions. Thus the heat transfer is rapid enough so that in the tests to date, the temperature of the sand near the desiccant bag has not risen above an apparent value of about 95°F. (room temperature about 75°F.).

This test has usually been applied to desiccant bags of 4.25 x 6 inches in inside dimensions with 3/8-inch heat seals on each side, and containing 140 grams of calcium oxide of 30 percent capacity and of about 70 to 90 percent volume increase on hydration.

After hydration is complete, the desiccant containers are examined for signs of rupture or damage to the heat seal or other closure.

Test. 2. Slow test under use conditions. The desiccant bags, with or without pinching-off as described above, are placed at the bottoms of No. 10 cans which are then filled with the product (in our test with 3 pounds of orange-juice powder). The cans are sealed and are then placed in storage in such a position that the desiccant bags are at the bottoms of the cans. After hydration is substantially complete (about 2 to 4 months for orange juice powder at room temperature) the cans are opened and the desiccant bags are examined for signs of rupture or damage to the seal.

This test under use conditions would be assumed to give results which are more reliable than those of the rapid empirical test, but the long time required for hydration limits the number of tests that can be made. Limited tests have indicated that bags may fail in the slow test even though satisfactory in the rapid test. A combination of the two tests may therefore be desirable: the rapid test as a guide in developing or testing desiccant containers and the slow test as a more reliable appraisal of the suitability of desiccant containers which show promise.



### III. Test of Calcium Oxide Desiccant Capacity and Reactivity

The following test was developed at this Laboratory as a rapid method of appraising the capacity and the reactivity of calcium oxide desiccant samples; it was included in the "in-can dehydration" specification (37) previously mentioned.

#### Equipment and reagents

- One or more 250-mm. desiccators with a sample support made of 1/4-inch-mesh wire screen or similar material (to replace the regular desiccator plate).
- Five hundred ml. of a saturated solution of pure sodium bromide with excess crystals in each desiccator, at 75°F. (24°C.).
- Two-ounce tin ointment boxes, 6 cm. in diameter, and 2 cm. in height, or suitable weighing dishes.
- Suitable temperature control at  $75 \pm 2^\circ\text{F.}$  ( $24 \pm 1^\circ\text{C.}$ ).

#### Procedure

- a. Weigh accurately 1.9 to 2.0 grams ( $\pm 0.005$  gram) of the ground sample into a sample container and spread it in a uniform layer over the bottom.
- b. Place the sample container in the desiccator. Use one desiccator for not more than six samples.
- c. Weigh the dish and sample after 48 hours and calculate the percentage of water absorbed by the desiccant from the increase.
- d. Weigh the dish and the sample after 48 hours (and if desired after successive intervals of 2 to 4 days until the weight becomes constant).
- e. Calculate the percentage of water absorbed by the desiccant from the increase in weight.

#### Tentative requirement

The desiccant should increase in weight at least 28.5 percent in 48 hours.

This test gives a composite measure of reactivity and of capacity; that is, for the sample to absorb water equal to 28.5 percent of its weight in 48 hours it must have both sufficient reactivity and sufficient capacity. Failure to absorb this quantity of water may be due either to low reactivity or to low capacity; the two can be differentiated by comparing the 48-hour value with the ultimate capacity (the moisture absorbed when the sample is held until constant weight is reached). From 4 days to 2 weeks or longer may be required to reach constant weight, depending on the sample.

If failure to meet the above requirement is due to low reactivity, there will be a large difference between the 48-hour capacity and the ultimate capacity, but if due to low capacity the difference will be small (for example, no more than about 2 percent). All the recalcined hydrated lime samples with which we have worked have been sufficiently reactive that the differences between



these values have never been more than 1 to 2 percent, although the ultimate capacity has sometimes been lower than 28.5 percent. Samples with capacity lower than 28.5 percent can of course be used for in-package desiccation. Proportionately larger amounts are required to remove a given amount of moisture from the product.

Samples of somewhat lower reactivity than required in this test could also be used for in-package desiccation, but the overall rate of desiccation will be somewhat reduced if the reactivity of the desiccant is much lower than that necessary for the ultimate capacity to be rather closely approached in 48 hours.

It may be noted that for this test the desiccators are at atmospheric pressure, whereas evacuation is recommended for some of the tests described above. The difference is due to the fact that in this test only 2 grams of desiccant are hydrated per sample, and the amount of water vapor required can be readily transmitted through the air of the desiccator at atmospheric pressure, but in the tests involving 100 grams of calcium oxide per sample, the desiccator must be evacuated if the much larger amounts of water vapor are to be transferred in a short time.

#### Literature Cited

1. Bruhn, C. A. Vorrichtung zum Trocknen oder Trocknenaufbewahren feuchter oder feuchtigkeitsempfindlicher Gegenstände. German patent 287,737. Jan. 16, 1914.
2. Burr, H. K., and Silveira, V. G. Stabilization of Dehydrated Cabbage by In-package Desiccation. Submitted to Food Technol. for publication. 1954.
3. Cotton, R. H., and Schroeder, A. L. Method of Preparing Products for Storage and Packaged Products Produced Thereby. U. S. Patent 2,520,878. Aug. 29, 1950.
4. Davison Chemical Corp. Product Data Sheet. Protek-Sorb 121. 1953.
5. Filtrol Corporation. Filtrol Alumina Product Bulletin No. 1, April, 1953.
6. Hendel, C. E., Bailey, G. F., and Taylor, D. H. Measurement of Non-enzymatic Browning in Dehydrated Vegetables During Storage. Food Technol. 4, 344-347, 1950.
7. Hendel, C. E., and Legault, R. R. Observations on Rate of In-package Desiccation. Food Technol. 8, 189-191, 1954.
8. Hartman, R. H. Trends in Dehydrated Packaging. Modern Packaging 26 (5):200-04, 1953.
9. Hockley, R. L. New Dehydration Packaging Method. Chem. and Engin News 21:2132, 2134-36, 1943.

10. Hockley, R. L. Keeping Goods Dry in Transit. Chem. and Met. Engin. 46:775-76, 1939.
11. Howard, L. B. Significance of Moisture Content in Dehydrated Vegetables. Processed. 12 pp. Presented at annual meeting National Dehydrators Assoc., Chicago, Ill., 1945.
12. Howard, L. B. Desiccants Improve Dry Packs. Food Packer 26(4):31, 1945.
13. Howard, L. B. Factors of Processing and Storage That Affect Quality. The Canner 100(13):46, 48, 50, 1945.
14. Johnson, C. M. Determination of Water in Dry Food Materials. Indus. and Engin. Chem., Anal. Ed. 17, 312-16, 1945.
15. Kahn, K. D. New Desiccant Keeps Quality in, Moisture Out. Food Indus. 20(1):86-7, 224-26, 1948.
16. Legault, R. R., Hendel, C. E., and Talburt, W. F. Retention of Quality in Dehydrated Vegetables Through In-package Desiccation. Food Technol. 8, 143-49, 1954.
17. Legault, R. R., Talburt, W. F., Mylne, A. M., and Bryan, L. A. Browning of Dehydrated Vegetables During Storage. Indus. and Engin. Chem. 39: 1294-99, 1947.
18. Legault, R. R., Hendel, C. E., Talburt, W. F., and Rasmussen, L. B. Sulfite Disappearance in Dehydrated Vegetables During Storage. Indus. and Engin. Chem. 41, 1447-51, 1949.
19. Legault, R. R., Hendel, C. E., Talburt, W. F., and Pool, M. F. Browning of Dehydrated Vegetables During Storage. Food Technol. 5, 417-23, 1951.
20. Lovett, R. A. New Uses for Desiccants. Modern Packaging 22(5):129-33, 1949.
21. Madaus and Co. Verfahren zum Trocknen und zur Lagerung wasserhaltiger, fluchtige Stoffe fuhrender Produkte. German Patent No. 656,890.
22. Makower, B., Chastain, S. M., and Nielsen, E. Moisture Determination in Dehydrated Vegetables--Vacuum Oven Method. Indus. and Engin. Chem. 38, 725-31, 1946.
23. Marc, C. Pate absorbante. French Patent No. 745,146, May 5, 1933.
24. McCorkhill, J. C. Moisture Absorbent. U.S. Patent No. 2,202,742, May 28, 1940.
25. Miller, R. N., and Robertson, W. E. Double Shelf Life--It's in the Bag. Food Engin. 23(7):38-39, 147, 1951.
26. Moyer, P. S. Means for Preserving Prepared Cereal, Cereal Products, and the Like. U.S. Patent No. 1,222,656, April 17, 1917.



27. \_\_\_\_\_. Improvements in or Relating to Moisture Absorbing Compositions. British Patent No. 205,173, July 10, 1922.
28. \_\_\_\_\_. Dehydrating Unit. U.S. Patent No. 1,614,753, Jan. 18, 1927.
29. Mylne, A. M., and Seamans, V. S. Stabilized Orange Juice Powder. II. Changes During Storage. Food Technol. 8:45-50, 1954.
30. National Research Council. International Critical Tables, 8:294. McGraw-Hill Co., New York, 1928.
31. Neusbaum, C. W. Container for Preserving Foods. U.S. Patent No. 1,657,272, Jan. 24, 1928.
32. Ohlhaver, P.D.H. A Process of Maintaining the Vitality of the Active Organisms in Dried Yeast. British Patent No. 27,573, Dec. 8, 1911.
33. \_\_\_\_\_. Process of Maintaining the Vitality of Dried Yeast. U.S. Patent No. 1,039,999, Oct. 1, 1912.
34. \_\_\_\_\_. Verfahren zur Aufbewahrung getrockneter Hefe unter Erhaltung ihrer Lebensfahigkeit. German Patent No. 257,177, March 2, 1911.
35. Strashun, S. I., and Talburt, W. F. WRRL Develops Techniques for Puffed Powder from Juice. Food Engin. 25(3):59-60, 1953.
36. Talburt, W. F., Hendel, C. E., and Legault, R. R. Desiccating in the Package. Food Engin. 26(4):79-81, 194, 197, 1954.
37. U.S. Army Quartermaster Corps. Tentative Specification CQD 384, Vegetables, Dehydrated; Packaging and Packing of, With In-Can Dehydration, Aug. 1945.
38. U.S. Army Quartermaster Corps. Tentative Specifications: Cabbage - CQD, 58C (1945); Carrots - CQD, 73D (1945); Onions - CQD 70D (1945); Potatoes, White - CQD, 59H (1945); Potatoes, Sweet - CQD, 60F (1945).
39. U.S. Dept. of Defense. Military Specification MIL-D-3464, Desiccants (Activated) in Bags; for Static Dehumidification and Packaging, April, 1951.
40. Wilkinson, H. H. New Package Dehydrator. Chem. and Engin. News 21: 1054-55, 1943.
41. Yule, W. T. British Patent, dated Jan. 28, 1845. Abs. in Jour. Franklin Institute, Ser. III, 11:179-80 (1846). 1845.





